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fuel were equivalent to those of petroleum fuels, the nitrogen oxides (NO<sub>x</sub>) were higher for the oil shale fuel. A high concentration of fuel bound nitrogen was implicated as the cause for the high NO<sub>x</sub> emissions. The oil shale derived fuel was found not to conform to specifications for contamination, existent gums, thermal stability, freeze point and viscosity at -34.5°C (-30°F). A program of post-refinery upgrading studies was initiated in order to improve these deviant properties. This program included filtration, distillation, clay and acid treatment and urea extraction. It was found that no one single post-refinery treatment could improve all deviant properties.

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TRENTON, NEW JERSEY 08628

NAPTC-PE-82

**MAY 1976**

EVALUATION OF A JP-5 TYPE FUEL DERIVED FROM OIL SHALE

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CONVERSION FACTORS: SI TO U.S. CUSTOMARY UNITS

<u>Convert From</u>	<u>To</u>	<u>Multiply by</u>
$\text{Kgw}^{-1}\text{s}^{-1}$	$\text{lb SHp}^{-1} \text{ hr}^{-1}$	$5.91835 \times 10^{+6}$
W	Hp	$1.34102 \times 10^{-3}$
$\text{Kgs}^{-1}$	$\text{lb hr}^{-1}$	$7.93664 \times 10^{+4}$
Pa	psi	$1.45037 \times 10^{-4}$
Nm	$\text{lb ft}$	$7.37562 \times 10^{-1}$
$t_{\circ}\text{C}$	$t_{\circ}\text{F}$	$t_{\circ}\text{C} = \frac{(t_{\circ}\text{F} - 32)}{1.8}$
$\text{JKg}^{-1}$	$\text{BTU lb}^{-1}$	$4.29922 \times 10^{-4}$
$\text{m}^2\text{s}^{-1}$	cks	$1.000000 \times 10^6$
litres	gallons	$2.64172 \times 10^{-1}$
litres	$\text{ft}^3$	$3.53146 \times 10^{-2}$
Kg	lb	$2.20462 \times 10^0$

## INTRODUCTION

Reference 1 authorized the implementation of Work Unit Plan 913 which included the testing and evaluation of a kerosene derived from oil shale as a substitute for current petroleum derived JP-5. In a separate program, the Office of Naval Research, via the Applied Systems Corporation, obtained a large quantity of refined shale oil (reference 2). In turn, the Applied Systems Corporation provided the Naval Air Propulsion Test Center (NAPTC) with 17,500 gallons of a kerosene (JP-5 type) fuel produced from the shale oil for evaluation purposes. The evaluation work in this program included the following: performance and exhaust emissions test on a T63-A-5A engine; JP-5 specification tests; in-house upgrading studies. The results of this work are presented in this report.

## CONCLUSIONS

1. The performance of the JP-5 type fuel derived from oil shale was equivalent to that of the petroleum derived JP-5 in the sea level operation of the T63-A-5A engine under the environmental condition tested.
2. The carbon monoxide (CO) and total unburned hydrocarbons (THC) emissions for the oil shale derived JP-5 were equivalent to those of the petroleum derived JP-5. The oxides of nitrogen (NO<sub>x</sub>) emission levels were higher for the oil shale derived JP-5. This is probably related to the high levels of fuel organic nitrogen compounds which are indigenous to oil shale.
3. The oil shale derived JP-5 failed to meet the Military Specification, MIL-T-5624J, for grade JP-5. The shale oil JP-5 contained a high degree of soluble and insoluble contamination. As a result of this contamination the fuel did not meet the requirements for existent gums and thermal stability. The freeze point and viscosity at -34.5°C (-30°F) were not within the specification limits. These latter deviations from specification requirements probably are the result of a high normal paraffin content of the fuel. The aromatic content was slightly higher than the allowable limit of 25 volume percent, but did not result in an unsatisfactory smoke point value.
4. There was no single post refinery upgrading technique which improved all deficient properties of the oil shale JP-5.
  - a. Redistillation eliminated solids contamination, almost eliminated existent gums and produced a thermally stable fuel. However, basic organic nitrogen was not removed from the fuel and freeze point was not improved.



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b. Clay treatment removed solids contamination, reduced existent gums, eliminated soluble metals, reduced basic organic nitrogen level and improved thermal stability. However, existent gums, thermal stability and freeze point still did not meet the limits of the specification. The large quantity of clay which would be necessary to remove most of the dissolved contamination is impractical for a commercial scale operation.

c. Acid treatment eliminated basic nitrogen, reduced existent gums and produced a thermally stable fuel. However, existent gums and freeze point still were not within specification limits.

d. Filtration reduced solids contamination temporarily.

e. Urea extraction improved the freeze point.

#### RECOMMENDATIONS

1. It is not recommended that the shale oil JP-5, whose properties are shown in this report, be used in flight operations. Furthermore, any additional engine testing should be delayed pending a source of supply of an acceptable quality of shale oil JP-5.

2. Further examination of the relationship between the level of basic nitrogen compounds and the thermal oxidation stability of shale oil kerosenes should be initiated.

3. Various laboratory tests should be initiated, on a low priority basis, to check other performance factors (i.e., material compatibility, cleanliness, equipment, additives, flammability characteristics) of the shale oil JP-5.

#### DESCRIPTION

##### T63-A-5A Engine Performance Test

1. The Allison T63-A-5A engine which was used for the performance and emissions evaluations is a turboshaft engine of the free turbine type. It is used in the Army OH-58A and Navy TH-57A helicopters. The gas producer section is composed of a combination six-stage axial flow one-stage centrifugal flow compressor directly coupled to a two-stage free turbine which is gas coupled to the gas producer turbine. The engine contains an integral reduction gearbox (5.84:1) which provides an internal spline output drive at the front of the gearbox. The engine has a single combustion chamber. The output shaft centerline is located below the centerline of the engine rotor and the exhaust is directed upward through dual exhaust pipes. An air bleed valve at the fifth compressor stage is provided to insure surge free accelerations.

2. The power turbine inlet temperature indication (T5) is provided by the average of four thermocouples located in the power turbine nozzle. The performance ratings of the T63-A-5A engine as specified in reference 3 are shown in Table I. The engine (Serial Number W-33) was supplied by NAPTC. Prior to this test, it had logged an undetermined number of hours since new and two hours since overhaul (June 1975).

3. The T63-A-5A engine was installed in a sea level test cell using a three-point mounting system. A flywheel and an Industrial Engineering Water Brake, Type 400 were connected to the engine gearbox assembly at the forward power output pad to absorb the engine power. The brake reaction was measured by a Baldwin load cell. All parameters to determine the engine starting and steady-state performance with the fuels were measured using standard test cell instrumentation. The engine was cleaned every 20 cycles (26.6 hours) with a twenty percent solution (by volume) of B&B 3100 and distilled water. It was also cleaned prior to the engine calibration with JF-5 fuel.

4. The following cycle, which is a modification of Specification MIL-E-8595 qualification cycle, was used for the performance test. The cycle was modified for a lube evaluation study that was being run concurrently with the performance test.

#### PERFORMANCE TEST CYCLE

<u>Engine Power Rating</u>	<u>Time (Minutes)</u>
Cold Start	
Ground Idle	2
Normal Rated	10
Ground Idle	2
Maximum Power	5
Normal Rated	55
Maximum Power	5
Ground Idle	1
Chop	
<hr/>	
TOTAL TIME	80

Engine inlet air and fuel temperature during the program was between 26.5 and 32.0°C (80° and 90°F).

Emissions Tests

5. The engine was not cleaned prior to the emission tests conducted on JP-5 and the synthetic fuel derived from oil shale.

6. The exhaust emission samples for JP-5 and oil shale derived fuels were taken at the following engine conditions, in sequence, for the pollutants measured:

<u>Engine Power Rating</u>	<u>Time (Minutes)</u>
Cold Start	--
Maximum Power (mil)	10
Normal Rated Power (NR)	10
90% NR	10
60% NR	10
40% NR	10
Flight Idle	10
Ground Idle	10
	<hr/>
TOTAL TIME	70

7. This sequence was then repeated to provide duplicate data. Throughout the test program, the power turbine (NPT) was kept at a constant speed of 538 RPS (35,000 RPM) except at ground idle. The engine power ratings designated for the emission survey were selected as being representative of a typical helicopter duty cycle. No engine bleed air flow was extracted from the engine during emission sampling except at ground idle and flight idle, when the compressor fifth stage acceleration bleed valve is automatically open. The compressor fifth stage acceleration bleed air flow is defined by the manufacturer in figure 60 of reference 3. The fuel-air ratio was calculated for each power rating with compensation made for the fifth stage bleed leakage at ground idle.

8. The instrumentation and methods of analysis for the engine emissions were in accordance with references 4 and 5.

9. The calibration gases used with the test instruments were purchased from Scott Research Laboratories and Matheson Gas Products. The specific gases used were:

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- a. CO - 1200 ppm, 890 ppm, 441 ppm, 250 ppm, 75 ppm, 25 ppm
- b. CO<sub>2</sub> - 4.74%, 3.0%, 2.0%, 1.0%
- c. NO + NO<sub>2</sub> - 190 ppm, 83.9 ppm + 6 ppm, 28.1 ppm + 2.6 ppm
- d. THC - 459 ppm, 408 ppm, 357 ppm, 200.4 ppm, 146.4 ppm, 36.6 ppm

The accuracy of all the above gases was guaranteed by the vendors to be  $\pm 1$  percent. The concentrations of these gases were controlled by availability and NAPTC needs, and do not exactly match the requirements in reference 4.

10. Emission sampling was done with a probe fabricated in accordance with references 4 and 5. Two probes were made because of the dual tailpipe configuration. They were made of stainless steel with four arms extending from a central manifold. The plane of each probe was an ellipse with major and minor axes of 231.8 mm (9-1/8 inches) and 177.8 mm (7 inches). There were three 1.524 mm (0.060 inch) diameter holes at the centers of equal areas on each arm. The probes were centered in the exhaust stream 41.3 mm (1-5/8 inches) downstream of the exhaust pipe exit. A probe was mounted in each exhaust pipe.

11. The emission sampling line was stainless steel with an internal diameter of 7.147 mm (0.305 inch). It was maintained at a temperature of  $150^{\circ}\text{C} \pm 5.0^{\circ}\text{C}$  ( $302^{\circ}\text{F} \pm 9^{\circ}\text{F}$ ). Nitrogen was blown back through the probe during engine start-up to preclude the deposition of raw fuel in the sampling lines.

### Specification Tests

12. On 21 April 1975, 17,500 gallons of a JP-5 type fuel derived from oil shale was received at the NAPTC fuel farm and standard fuel storage procedures were initiated. The crude shale oil was produced in a pilot plant at Anvil Points, Colorado which is operated by the Paraho Development Corporation. Refining of the crude shale oil was undertaken by Applied Systems, Incorporated via a contract let by the Office of Naval Research. Details of the production and refining of this batch of JP-5 type fuel are given in reference 2.

13. A sample of the shale oil JP-5 fuel was subjected to laboratory analyses to determine its conformance to Military Specification MIL-T-5624J for JP-5 fuel. All analytical tests were performed in accordance with ASTM Standard Methods as set forth in the 1974 ASTM Annual Book of Standards (volumes 23, 24, and 25).

### In-House Upgrading Studies

#### Filtration

14. The 17,500 gallons of shale oil JP-5, when received, was first

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passed through standard fuel filters to remove dirt and water. Severe plugging and high pressure differentials were recorded across the filters. A series of fuel filtrations were performed using standard paper element 10 micrometer filters in an effort to remove solid contamination.

15. In a separate study a sample of shale oil JP-5 was filtered through a Millipore Corporation 0.45 micrometer filter and allowed to stand at room temperature. Solid contamination levels were determined on the filtered fuel sample (ASTM Method D-2276) at intervals of 0, 3, 6, and 14 days storage.

### Distillation

16. Distillation of the shale oil JP-5 was performed on one liter batches through all glass apparatus at atmospheric pressure. The volume and temperature of distillate cuts were recorded. In one experiment two cuts were made on the distillate (initial boiling point (IBP) to 232.0°C (450°F); 232.0 to 255.5°C (450 to 492°F)) and basic nitrogen was quantitatively determined on each fraction (reference 6). In a second experiment, the fuel was distilled and fractions of 10 volume percent each were collected. The freeze point was determined for these fractions separately and in combination. Then the distillation cut-off temperature which would provide an acceptable freeze point for the shale oil JP-5 was determined.

### Clay Treatment

17. In an effort to improve fuel properties such as existent gums and thermal stability the shale oil JP-5 was subjected to adsorption chromatography over Attapulugus clay. The clay, which was supplied by Indiana Commercial Filters Corporation as 60/80 mesh was used directly without pretreatment. The clay was dry packed to a volume of 1.0 litre in a glass column 10 centimetres in diameter. The column was charged with 10 litre of shale oil JP-5 and the fuel was allowed to percolate by gravity through the clay. The first, through ninth and last, 10 volume percent of effluent fuel were subjected to laboratory analysis to determine existent gum content, contamination, thermal oxidation stability, basic nitrogen and soluble metals content.

18. The quantity of fuel which overloads the clay was determined by measuring the light transmission at 540 nanometres of successive aliquots of effluent fuel on a Bausch and Lomb Spectronic 20 spectrophotometer.

19. In another experiment the shale oil JP-5 was introduced into a single pass clay treatment unit which used 12.7 Kg (28 pounds) of clay to treat 20.4 Kg (45 pounds) of fuel. A schematic of the clay treatment unit is shown in figure 1. After treatment, the fuel was collected and the percent light transmission for an aliquot was determined. From light transmission data the effectiveness of the clay at removing solids could be determined. The initial volume of fuel was passed through the clay five times. After the fifth pass, the existent gum content and thermal stability of the treated fuel were determined.

#### Acid Treatment

20. The shale oil JP-5 was treated with mineral acid in an effort to reduce the existent gums and improve the thermal oxidation stability. The shale oil JP-5 (700 ml) was placed in a separatory funnel and extracted with 50 percent sulfuric acid solution (35 g). The fuel was then washed with tap water, dilute sodium bicarbonate and finally water. The fuel was then passed through one-tenth its volume of Attapulugus clay (60/80 mesh; no pretreatment) to remove any fuel impurities introduced by the acid or water washes. The fuel was then dried ( $\text{Na}_2\text{SO}_4$ ), filtered and subjected to analysis for the determination of existent gums content, thermal oxidation stability, freeze point, and basic nitrogen level.

#### Urea Extraction

21. The higher freeze point of the shale oil JP-5 was attributed to the very high content of normal paraffins in the fuel (see Results and Discussion). In an effort to reduce the normal paraffin content the shale oil JP-5 was treated with urea. The method is outlined below:

22. In a large beaker, 100 g of shale oil JP-5, 100 g urea (Merck, Reagent Grade) and 300 ml of cyclopentane or petroleum ether (boiling range 30-60°C (86-140°F), Reagent Grade) were mixed by stirring for five minutes. Methanol (97 percent) was added in five ml portions (40 ml total) at five minute intervals with stirring. The mixture was stirred for one hour then filtered. The filter cake was washed well with petroleum ether. The petroleum ether was removed from the combined filtrate by evaporation on a rotary evaporator at 30°C (86°F). The urea extracted fuel was subjected to analysis to determine distillation curve, flash point, freeze point, hydrocarbon type and viscosity (at -34.5°C (-30°F)). The crystalline urea-inclusion compound was isolated and the included paraffins obtained by dissolving the crystals in hot water and separating the organic layer. Gas-liquid partition chromatographic (glpc) analysis was performed on the isolated extracted paraffins by Dr. R. N. Hazlett, Naval Research Laboratory (NRL), using a capillary column and electronic integration.

## RESULTS AND DISCUSSION

### T63-A-5A Engine Performance

1. Prior to the T63-A-5A engine performance and exhaust emission tests, calibration runs were made with a conventional JP-5. Analysis (Table II) showed this fuel to conform in all respects to the MIL-T-5624J specification. A graph of shaft power (SP) versus the power turbine inlet temperature, °C (°F), is shown in figure 2. The guarantee model specification requirements, reference 3, for the T63-A-5A engine are also shown for comparison purposes. It can be seen that the T63-A-5A, using a conventional petroleum derived JP-5, conforms to the model specification requirements.

2. The performance of the shale oil JP-5 was found to be equivalent to the base line JP-5 as is also shown in figure 2. The corrected engine data are shown in Table III. Although this fuel was highly contaminated with solid particles (see Fuel Analysis section) no effect on engine performance could be discerned. Most of the solid matter was collected at the two in line filters and at a filter just upstream of the engine fuel pump. In figure 3, a photograph of these fuel filters shows the removal of particulates as the fuel approaches the engine.

### Exhaust Emissions

3. The exhaust emission levels of CO, unburned hydrocarbons and oxides of nitrogen for the conventional JP-5 as well as the shale oil JP-5 are shown in figures 4, 5, and 6. The emission levels in these figures are in parts per million (ppm) as a function of fuel air ratio. The shale oil JP-5 gave equivalent carbon monoxide and unburned hydrocarbon emissions compared to the petroleum derived JP-5. However, the shale oil JP-5 produced higher oxides of nitrogen levels than the conventional fuel at all fuel-air ratios examined. It is possible that nitrogen compounds in the fuel caused higher NO<sub>x</sub> emission levels (see Fuel Analysis section).

### Fuel Analysis

4. A sample of the shale oil JP-5 was subjected to analysis to determine its conformance to the MIL-T-5624J specification for grade JP-5. The data, presented in Table IV, shows that while the shale oil JP-5 conforms to many of the specification requirements, it diverges markedly from the requirements for contamination, freeze point, existent gums and thermal oxidation stability. The shale oil fuel also possessed a dark color and foul odor. Color and odor are not current specification requirements, however, color can be an indicator of a highly oxidized and therefore unstable fuel. The odor was that of amines and raises the question of personnel safety. Specifically the toxicity and carcinogenicity of amines in the JP-5 boiling range are, at present, unknown. In addition certain nitrogen containing compounds are known to induce deposit formation in kerosene fuels (reference 7).

5. The sample of shale oil JP-5 received at NAPTC would not be an acceptable substitute for petroleum derived JP-5. This sample was to be used in the performance and exhaust emissions test of the T63-A-5A engine. However, the contamination levels were so high as to preclude the use of the shale oil fuel until the fuel contamination problem could be corrected. Since this fuel was high in gums, had a poor freeze point and did not meet thermal stability requirements, a program was initiated to examine a number of post-refining approaches for up-grading the shale oil JP-5 which included: filtration, distillation, clay treatment, acid treatment and urea extraction.

#### Filtration

6. The shale oil JP-5 which was received at NAPTC on 21 April 1975 was first transferred from the delivery truck into large fuel storage tanks. All fuels which are received and stored in this manner are first filtered. The shale oil JP-5 caused a pressure drop of 310 kPa (45 psi) across the filter. Under normal conditions a pressure drop of 103 kPa (15 psi) can be expected only after several hundred hours of use. After one truck was emptied, the filter was changed. The spent unit was examined and a black tarry mass was found on the filter (figure 7a). This tarry residue has the appearance of crude shale oil. It was evident from contamination values for the filtered fuel that the filter did not stop this material completely. It seems probable that the tar squeezed through the filter under the excessive pressure.

7. An attempt was made to filter the fuel in the following manner: eight 10 micrometer filters were series connected in a line between an empty clean storage tank and the tank containing the contaminated shale oil JP-5. The shale oil JP-5 was pumped through the filters into the clean tank. Samples obtained before and after filtration, were analyzed for solid contamination. The contamination level of the fuel obtained just prior to filtration revealed that most (90 percent) of the solids had settled to the bottom of the storage tank. In addition, those solids which remained suspended were not stopped by the 10 micrometer filters (see Table below). The tank

<u>Date</u>	<u>Contamination, mg l<sup>-1</sup></u>	<u>Remarks</u>
21 April 1975	164.2	"As received" sample.
7 May 1975	13.3	Sample just prior to filtration.
7 May 1975	13.3	Filtered fuel; 98 volume percent filtered.

bottoms (last 2 volume percent) were then inadvertently pumped through the eight series-connected filters. The high instantaneous pressure drop caused each filter element to rupture, thereby contaminating the filtered fuel. This tank was allowed to stand for a few days and 98 volume percent



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of the fuel transferred to another tank. The transferred fuel had a contamination level of  $7.9 \text{ mg l}^{-1}$ . This fuel was then used for engine performance and exhaust emissions testing.

#### Solids Deposition Rate

8. After transferring the shale oil JP-5 into a clean tank, samples were taken over several weeks time and contamination levels measured. The contamination versus time data are shown in the Table below:

<u>Date</u>	<u>Storage Tank Contamination, <math>\text{mg l}^{-1}</math> (0.35 mm (14") from Bottom of Tank)</u>
19 May 1975	7.9
2 June 1975	28.4
25 June 1975	30.1
27 June 1975	37.8
14 July 1975	31.8

The data show that the shale oil JP-5 is quite unstable and solid particulate matter (sediment) is continuously forming. The sediment which forms upon storage is quite different in physical appearance from that tarry matter initially collected from the fuel farm filter (figure 7a). It is this "new" sediment which is only partially stopped by 10 micrometer fuel filters (see figure 3). Hence, filtration of this batch of shale oil JP-5 results in lowered fuel contamination levels but only for a short period of time.

#### Distillation

9. Filtration of the shale oil JP-5 can lower the contamination level of the fuel, but existent gum and basic nitrogen as well as thermal oxidation stability and freeze point remain unaffected. A sample of the shale oil JP-5 was redistilled in an effort to improve these properties. The fuel was redistilled in two runs and a different distillation end point was employed for each run. As shown in Table V, some of the above fuel properties can be improved by distillation. Existent gum values and thermal oxidation stability are markedly enhanced by redistillation. Basic nitrogen compounds appear to be more concentrated in the lower boiling fraction of the fuel while those compounds which cause thermal oxidation instability are concentrated in the last 10 volume percent (probably in the distillation residue).

10. The change in freeze point with changes in distillation end point was carefully examined in order to determine the optimum fractionation

point which would provide a fuel with a freeze point of  $-46.0^{\circ}\text{C}$  ( $-51^{\circ}\text{F}$ ). In one experiment the shale oil JP-5 was redistilled and 10 volume percent fractions of distillate were collected (one cut was 15 volume percent (see figure 8)). The corresponding boiling range of each distillate fraction was recorded. The freeze point of each fraction was then determined. As shown in figure 8, approximately 70 volume percent of this fuel freezes above  $-46.0^{\circ}\text{C}$  ( $-51^{\circ}\text{F}$ ). However, the freeze point of a fuel is sensitive to its total composition. Hence the interaction of compounds in the high freeze point fractions could result in a lowering in freeze point when these fractions are recombined. In order to obtain an estimate of how much fuel could be redistilled while maintaining a freeze point of  $-46.0^{\circ}\text{C}$  ( $-51^{\circ}\text{F}$ ) the following experiment was performed. Another sample was distilled and individual 10 volume percent distillate fractions similar to those which are depicted in figure 8, were sequentially (10 + 20; 10 + 20 + 30; etc.) recombined and the freeze point determined for each combination. The results are presented graphically in figure 9. The distillation end point for each of the recombined fractions shown is that of the highest individual fraction. As shown in figure 9, redistillation of the shale oil JP-5 must be terminated at  $229.5^{\circ}\text{C}$  ( $445^{\circ}\text{F}$ ) to achieve a freeze point of  $-46^{\circ}\text{C}$  ( $-51^{\circ}\text{F}$ ) for the distillate (fuel). This distillate would represent approximately 55 percent of the original shale oil JP-5.

#### Clay Treatment

11. The sludge found on the filter-separator unit (figure 7a) was not the only source of contamination of the shale oil fuel. A soluble residue was isolated from the shale oil by column chromatography over activated silica gel (methanol elution) (figure 7b). It is this fuel soluble material which probably is responsible for the poor thermal stability of the fuel. In commercial operations, Attapulugus clay is used as a chromatographic adsorbent to separate soluble contaminants in fuel such as those shown in figure 7b.

12. A small quantity (10 litres) of shale oil JP-5 was percolated through clay as described earlier (Description, paragraph 17). It will be noted that clay treatment did have some effect on improving fuel quality (Table VI). This was evidenced by a 100 percent removal of the tar-like solid contamination, soluble metals and basic nitrogen in the first 1:1 (fuel:clay) effluent (10 volume percent). Other improvements detected were a 50 percent reduction in the gum content and a  $19.5^{\circ}\text{C}$  ( $35^{\circ}\text{F}$ ) increase in the thermal oxidation stability. The last 10 volume percent of effluent (a 9:1 fuel to clay volume ratio) shows overloading of the capacity of the clay. The basic nitrogen and gum contents are equivalent to those values before clay treatment. The thermal stability improvement decreased to  $240.5^{\circ}\text{C}$  ( $465^{\circ}\text{F}$ ), only  $8.5^{\circ}\text{C}$  ( $15^{\circ}\text{F}$ ) above the breakpoint temperature of the untreated fuel. The solid contamination and soluble metals, however were still being removed by the clay. Obviously the clay was acting as a filtering as well as an adsorbing medium which removed the suspended tar-like particulate matter and some gums. The overall effects expected of a clay-treatment-process on shale oil fuel are reflected in a composite (recombination) of all the fractions. The

composite is representative of clay treated shale oil JP-5 at a 9:1 volume ratio of fuel to clay. The analysis of the composite shows improvement in gum content (50 percent removal), and an increase in the thermal oxidation stability of 14.0°C (25°F) over the untreated fuel. These improvements, however, do not bring the fuel within specification limits for the latter two requirements.

13. To obtain an idea of the clay to fuel ratio where the clay became overloaded, light transmission measurements were made of the effluent fuel. Color is neither a specification requirement nor a major criterion for determining fuel quality. However, it is generally employed to ascertain the existence of dissolved high molecular weight material or contamination. In the case of the shale oil JP-5, color reduction by clay treatment has been used as a method to detect the removal of dissolved crude and cracked stock. In figure 10 a plot of the light transmission of the clay treated fuel as a function of the throughput is shown. At a low shale oil JP-5 throughput, very little light absorption is observed. The volume ratio of fuel to clay that is effective in reducing contamination is 1:1. However, beyond this ratio color bodies begin to appear quite rapidly due to saturation of the active sites on the clay. To illustrate how impractical it would be to employ clay treatment as a method of upgrading this shale oil JP-5, a comparison with a full scale commercial operation is in order. To clay treat 1.506 m<sup>3</sup> (12 barrels) of synfuel at a 2:1 volume ratio (fuel to clay) 1133 kg (1.25) tons of clay would be required. A typical commercial process employs fuel:clay ratios of 8.27-40.86 m<sup>3</sup> (52-257 bbl):907 Kg (1 ton). Hence a commercial unit would require between 5 and 27 times the quantities of clay normally used to effectively treat this shale oil.

14. Since percolation of the fuel over clay may not be the most effective method of clay treatment, another technique was evaluated. This technique involved circulating the shale oil through a single-pass small scale clay-treating unit (figure 1). Five passes of the shale oil JP-5 were used for the initial evaluation. The data are presented in Table VII. It can be seen from these data that a multiple pass through clay does not improve the quality of the fuel significantly with respect to the existent gum and thermal oxidation stability. An additional run was made of the previously clay treated fuel using fresh clay. This now represents a 4.6:1 volume ratio of shale oil JP-5 to clay. The analytical data from this second treatment with respect to the percent light transmission, gum content and thermal oxidation stability are also given in Table VII. Improvement in shale oil properties was not obtained and it may therefore be concluded that full scale clay treatment would not be a practical method of achieving conformance with the MIL-T-5624J fuel specification for this batch of shale oil.

#### Acid Treatment

15. Sulfuric acid treatment of fuel is known to remove resinous and asphaltic substances which may be left in fuel because of poor refining practices (reference 8). Sulfuric acid treatment also removes, to varying extents, compounds such as alcohols, aldehydes, ketones and naphthenic acids which are primary products of air autoxidation.

Nitrogenous bases such as amines, anilines, quinolines and pyridines are also removed by treatment with dilute acid. Since the shale oil had all the characteristics of a poorly refined fuel it was anticipated that sulfuric acid treatment would greatly improve the fuel.

16. Two samples of shale oil JP-5 which differed in the level of contamination were chosen for sulfuric acid treatment experiments. The "as received" fuel (see Table IV for analysis) and the effluent from the clay treatment experiments were chosen. The latter fuel was the composite effluent at a fuel:clay ratio of 9:1 (see Table VI). The fuels were treated with sulfuric acid as described above (see Description, paragraph 20). The acid treated fuels were subjected to analysis for existent gum, basic nitrogen level, thermal oxidation stability and freeze point determination and the results are presented in Table VIII.

17. It will be noted that acid extraction succeeded in removing all basic nitrogen and improved the thermal stability of the fuel. However, the existent gum levels were still above the specification requirement. This is somewhat surprising since gums are generally believed to be advanced products of fuel autoxidation. The freeze point was not improved but rather deteriorated as a result of acid treatment. Apparently the basic nitrogen compounds present lend solvent character to the fuel and act as mild freeze point depressants. Lastly, the color and odor of the acid treated fuels were equivalent to those of a conventional JP-5.

#### Urea Extraction

18. It has been shown that by simple post refining treatment steps such as filtration, distillation, clay and acid treatment, the poor properties of the shale oil JP-5 could be improved. Only the freeze point problem resisted resolution. These results imply that the high freeze point was caused by a fundamental chemical difference of shale oil JP-5 compared to petroleum derived fuels.

19. In a coordinated Navy research program the shale oil JP-5 was subjected to analysis by capillary column gas chromatography at the Naval Research Laboratory, Washington, DC (reference 9). Normal (straight chain) paraffins usually appear as sharp peaks in fuel mixtures and are therefore easily distinguishable. In Table IX are shown the results of glpc analysis for normal paraffins for the shale oil JP-5. The shale oil JP-5 contains over 36 percent normal paraffins. Almost 11 percent of the total shale oil JP-5 consists of  $n-C_{14}$ - $n-C_{16}$  paraffins. These paraffins have extremely high freeze points and low solubility in aromatic fluids. The data clearly show that normal refinery or inexpensive post refinery treatment will not improve

the freeze point of the fuel.

20. It is well known, however, that urea,  $(H_2N)_2CO$ , forms inclusion compounds (reference 10) with normal paraffins and that this property of urea forms the basis of commercial dewaxing processes (reference 11). Thus if selective removal of the  $n-C_{14}$ - $n-C_{16}$  paraffins could be effected a large improvement in freeze point and minimum loss of fuel yield would be realized.

21. The shale oil JP-5 was subjected to urea "dewaxing" treatment. The yield of fuel after urea treatment was approximately 82 percent (weight basis). Some physical properties of this fuel are presented in Table X. The anticipated reduction in the freeze point was realized. The viscosity of the urea treated fuel is also well within military specifications for grade JP-5 fuels. Thus urea treatment is much more efficient in producing a low freezing shale oil JP-5 than redistillation, because of the selective removal of high freeze point normal paraffins by urea. This can be easily seen from the data presented in Table XI. The urea included compounds were isolated and analyzed by capillary column gas chromatography. Almost 98 percent of the urea extracted material was found to be normal paraffins. The small quantity of normal pentane (from petroleum ether used; see Description, paragraph 22) may be removed by redistillation. The "percent extracted" column represents the fraction of normal paraffin removed from the original shale oil JP-5 by the urea treatment. Optimization of the fuel yield while maintaining a  $-46.0^\circ C$  ( $-51^\circ F$ ) freeze point for the urea extraction treatment was not attempted.

#### Summary

22. The shale oil JP-5 received by NAPTC on 21 April 1975 is not an acceptable substitute for conventional petroleum-derived fuel. While there is no detectable performance difference between shale oil JP-5 and petroleum JP-5 (T63-A-5A performance test), the  $NO_x$  emissions of the shale oil JP-5 were significantly higher. Presumably the presence of 900 ppm basic nitrogen compounds in the fuel account for the increased  $NO_x$  emissions. In addition, many MIL-T-5624J specification requirements were not met for the shale oil JP-5. The high contamination and gums could plug fuel lines in a short period of time. The poor thermal stability precludes the use of this fuel as a heat sink on aircraft. The high freeze point prohibits the use of this fuel in cold environments and possibly at altitude.

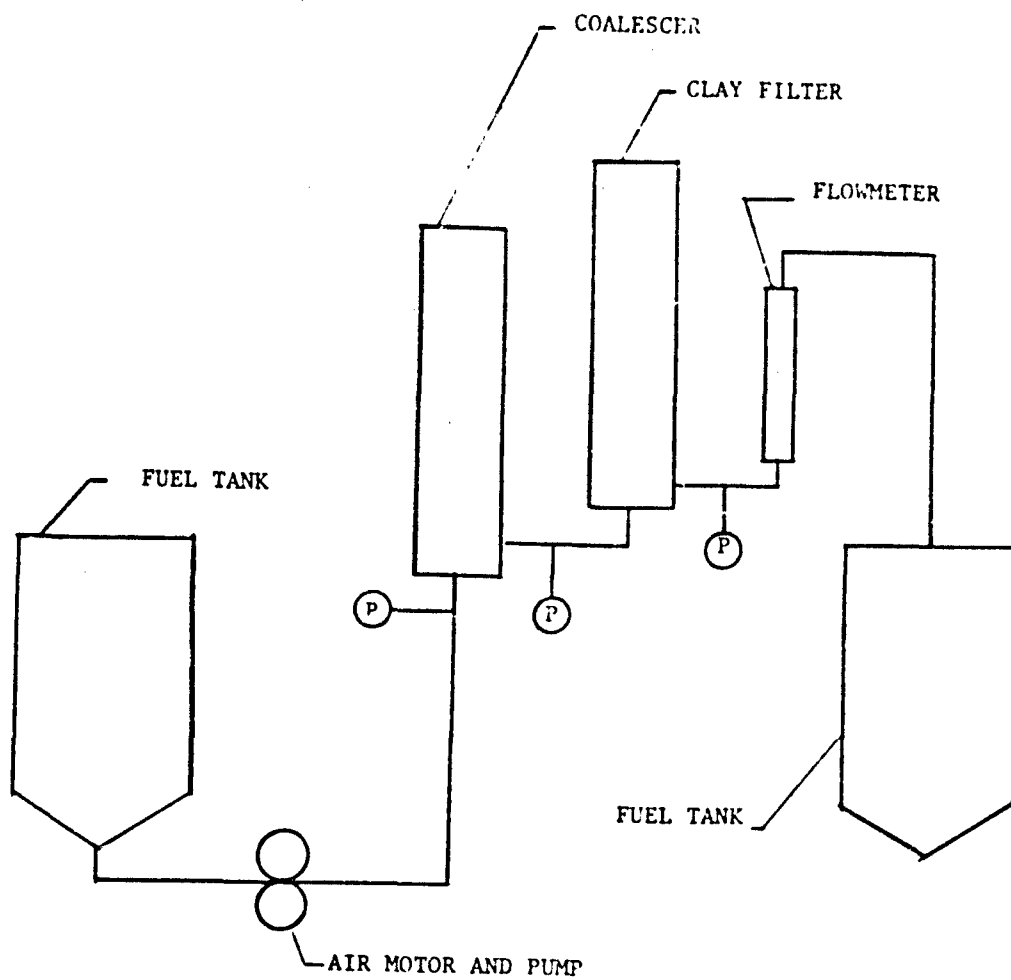
23. There are post-refinery treatments which succeed in improving some of the poor properties of this fuel. Filtration will reduce the contamination level but only for a short period of time. Existent gum levels can be reduced by distillation with a loss in fuel yield of about 10 percent. Thermal oxidation stability can be improved by either distillation or acid treatment. The former treatment incurs a 10 percent loss of fuel. The basic nitrogen level, which is not a MIL-T-5624J requirement, can be reduced only by acid treatment. The freeze point can be improved only by removal of normal paraffins from the fuel. In order to achieve a  $-51^\circ F$  freeze point a fuel loss

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of the order of 18 percent will be suffered. Thus, the technology is available to produce a specification fuel from the sample of shale oil JP-5 received at NAPTC. However, this will probably result in a loss of approximately 30 volume percent of original fuel.

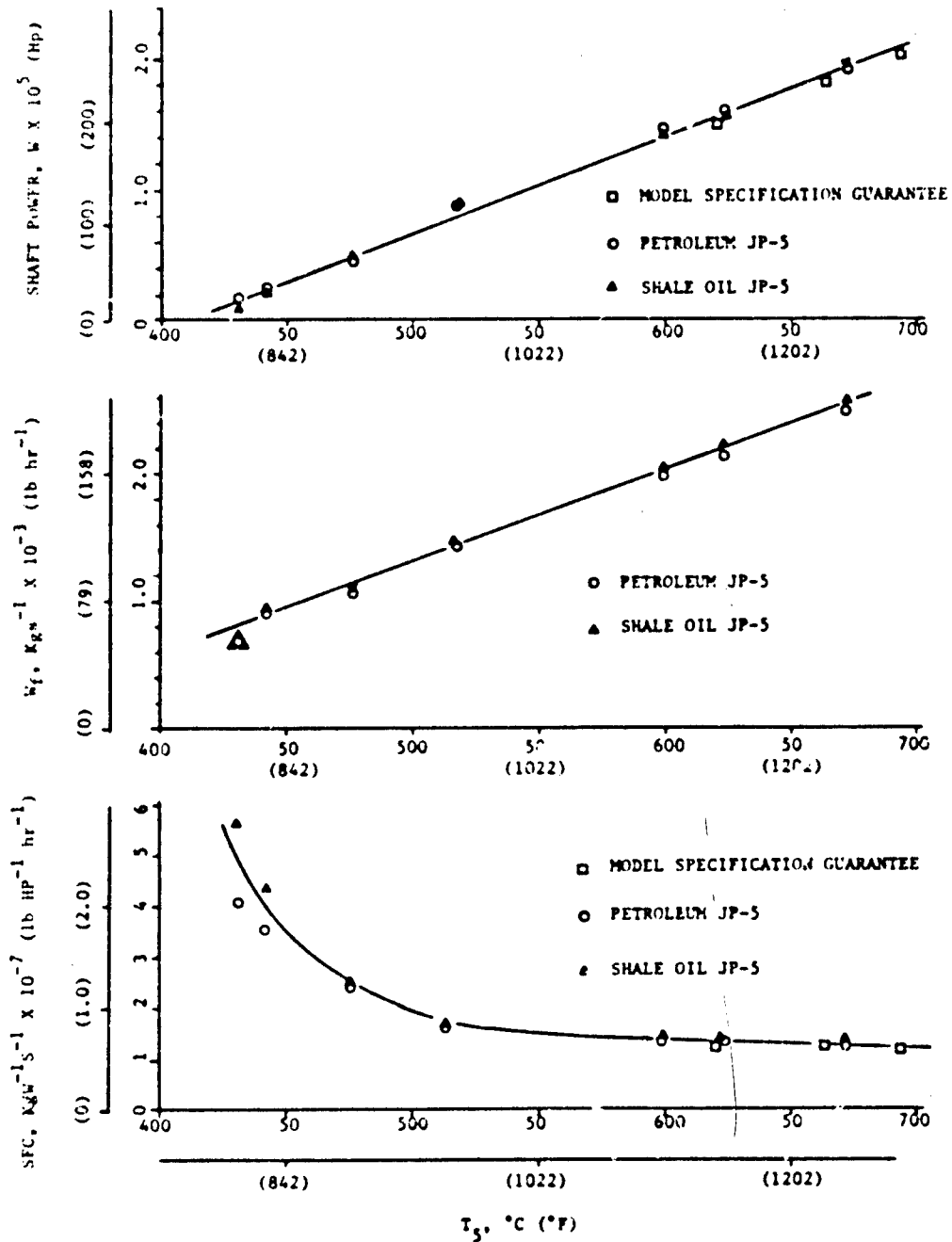
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FIGURE 1. SCHEMATIC OF OPEN-LOOP CLAY TREATMENT UNIT



(P) PRESSURE GAGE

FIGURE 2. T61-A-5A ENGINE PERFORMANCE OF SHALE OIL JP-5





COPY AVAILABLE TO DDC DOES NOT  
PERMIT FULLY LEGIBLE PRODUCTION

FIGURE 3. IN-LINE FUEL FILTERS USED DURING T63-A-5A ENGINE TEST WITH SHALE OIL JP-5

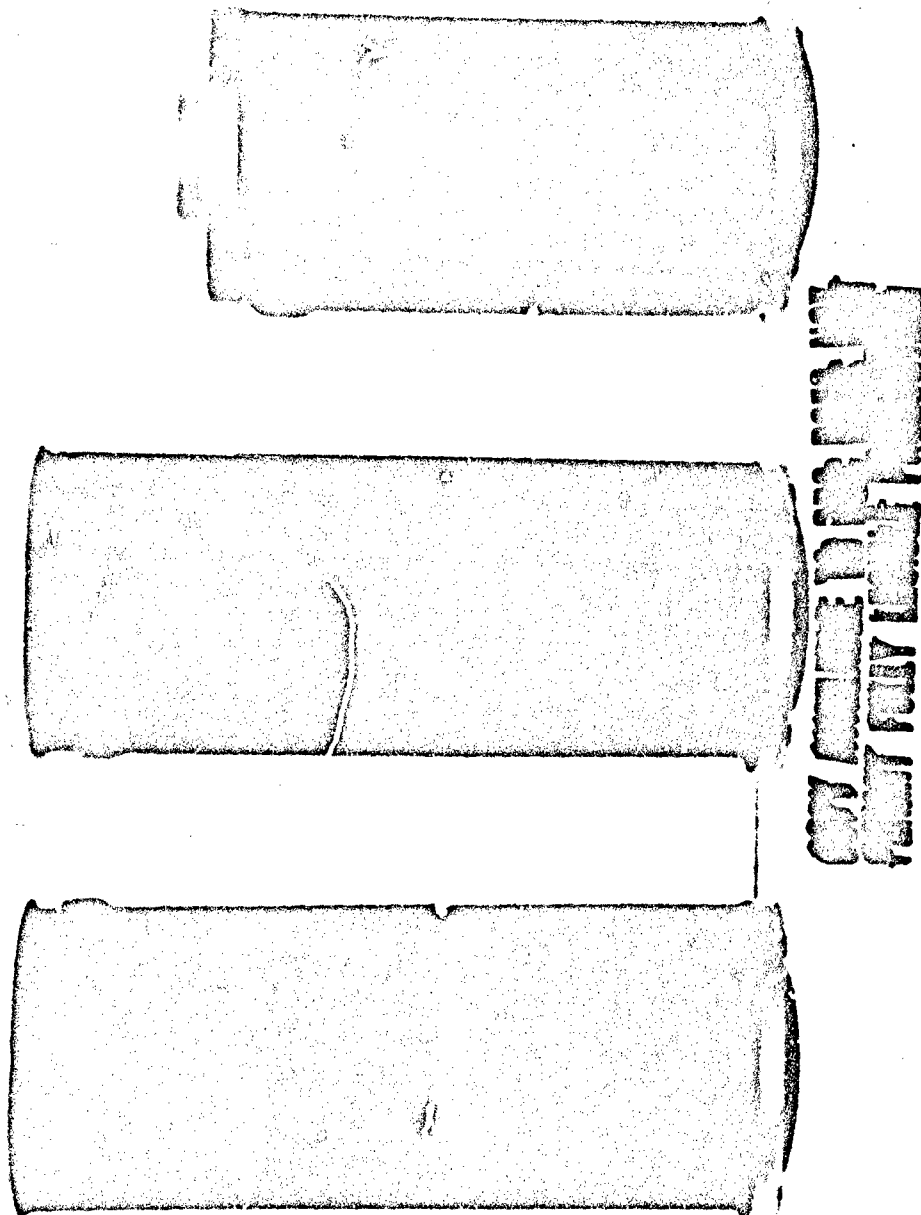


FIGURE 4. EXHAUST EMISSIONS OF CARBON MONOXIDE (CO) FOR T63-A-5A ENGINE

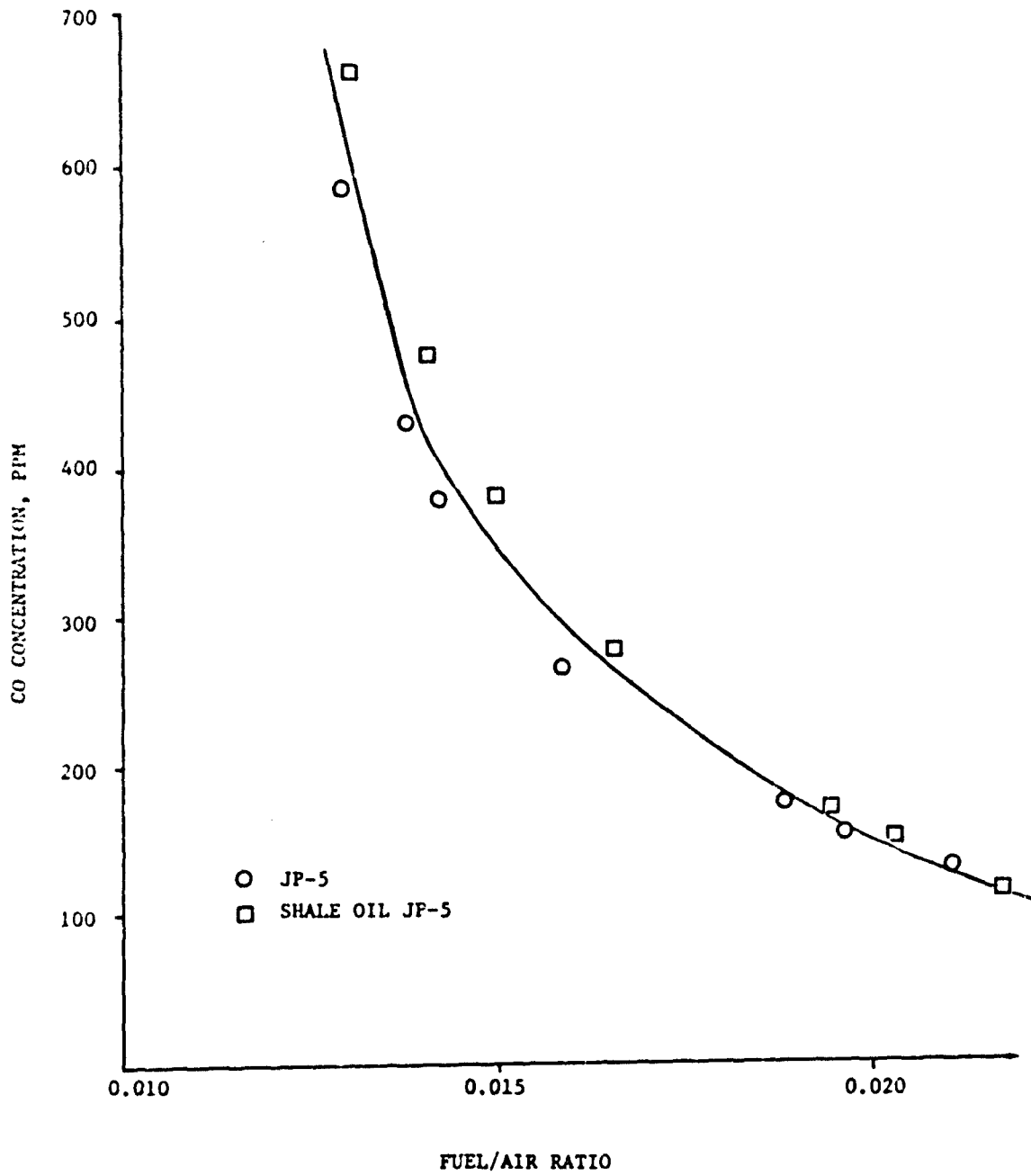


FIGURE 5. EXHAUST EMISSIONS OF TOTAL UNBURNED HYDROCARBONS (THC) FOR T63-A-5A ENGINE

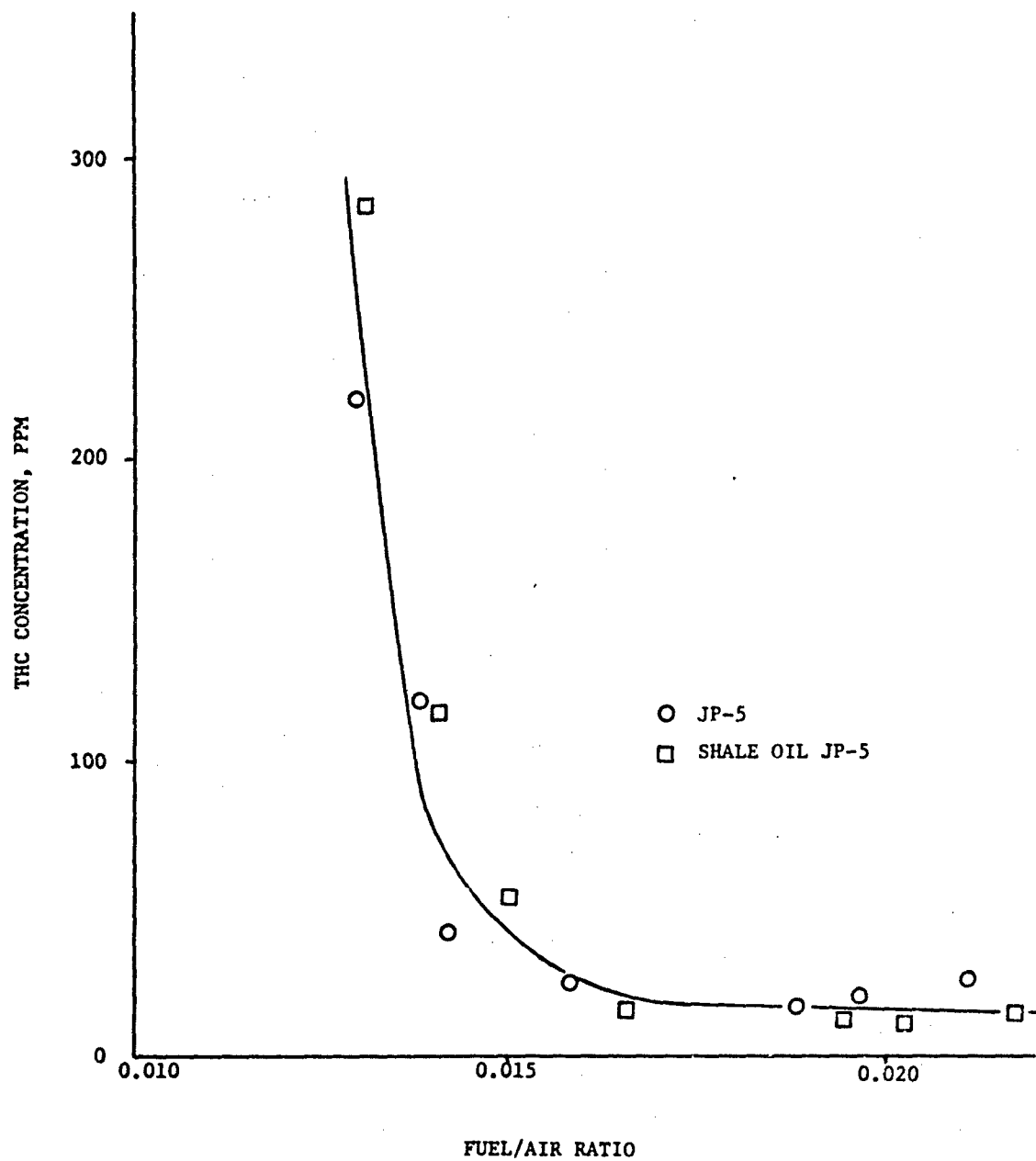


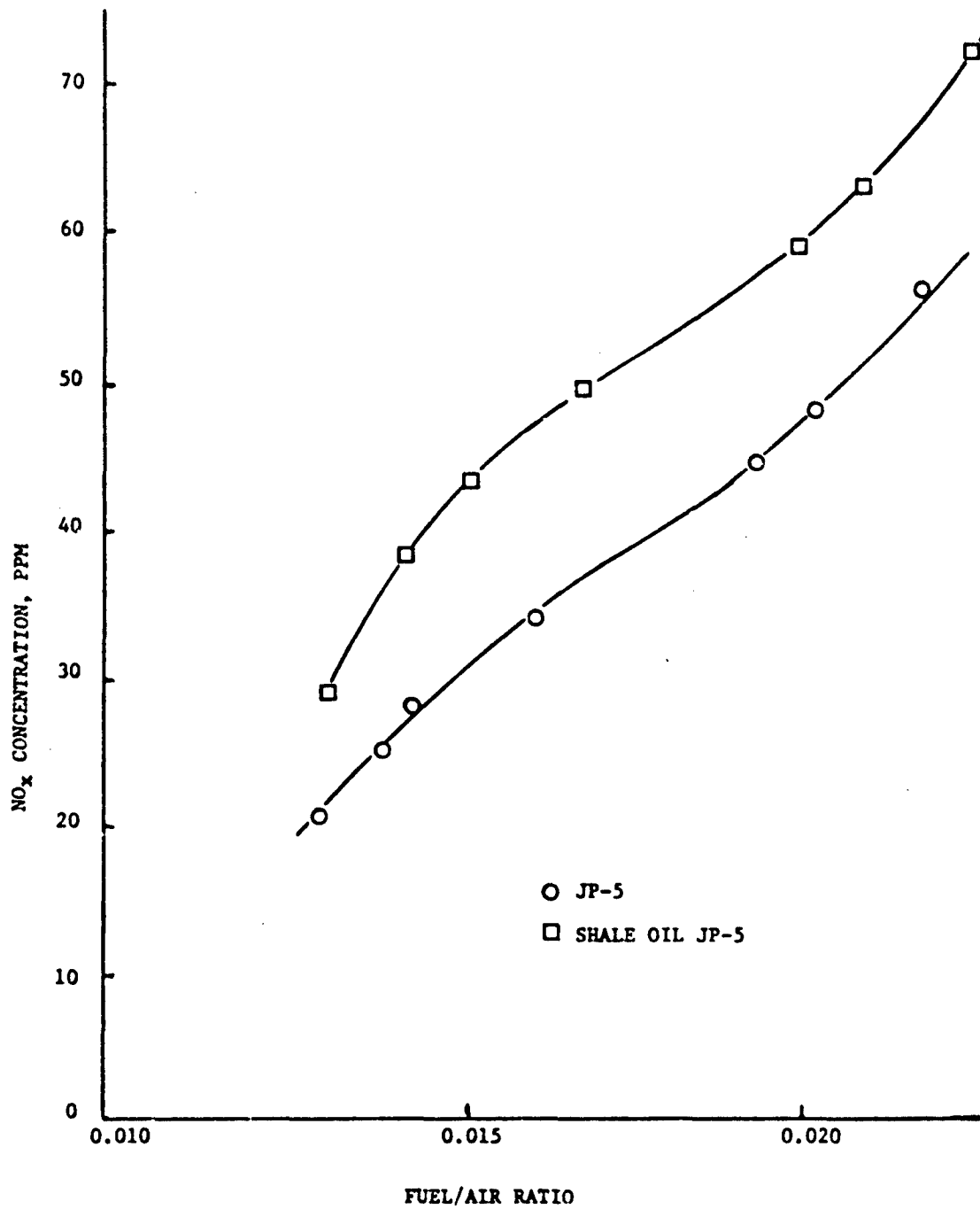
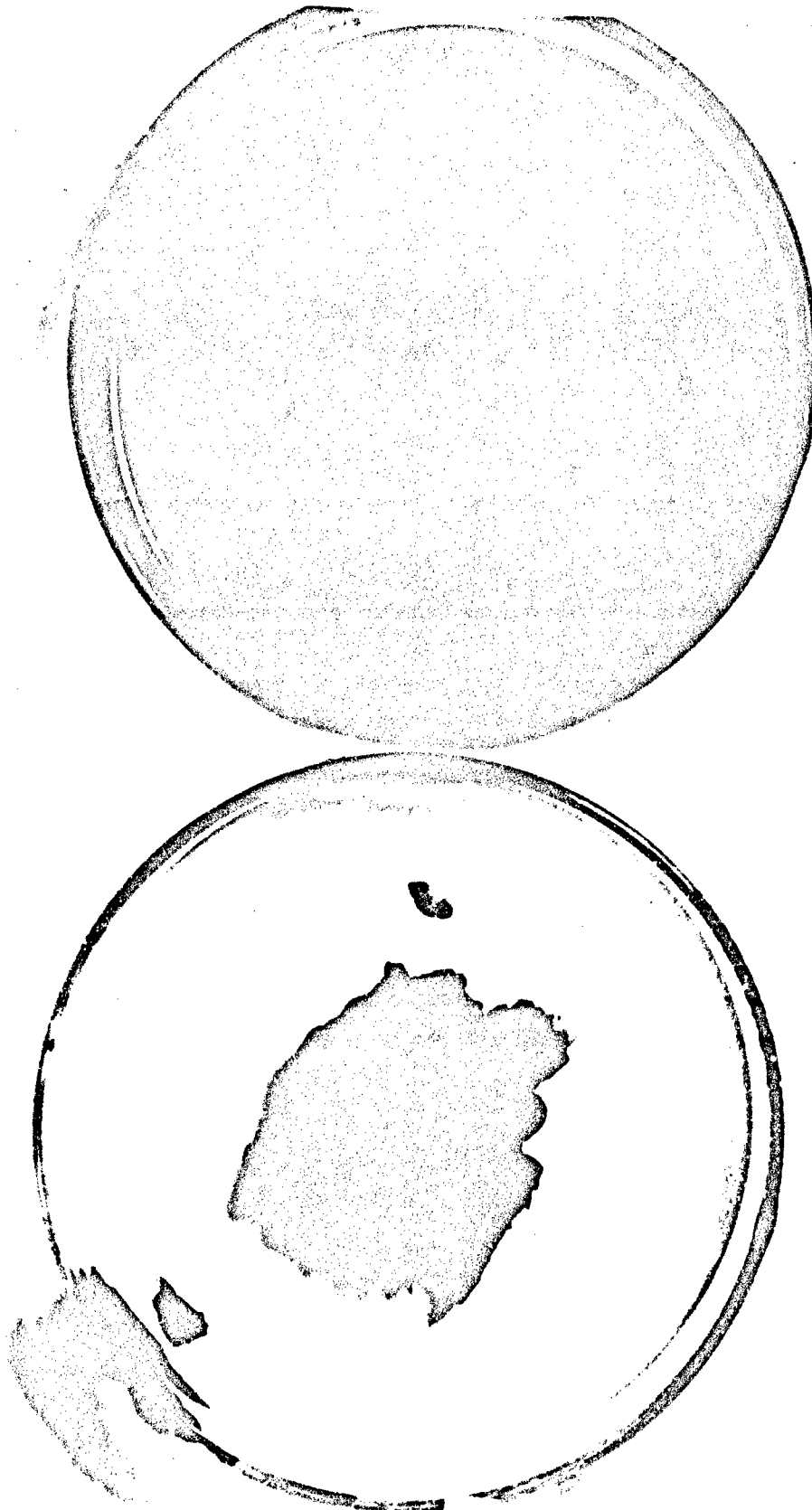
FIGURE 6. EXHAUST EMISSIONS OF OXIDES OF NITROGEN ( $\text{NO}_x$ ) FOR T63-A-5A ENGINE

FIGURE 7. SHALE OIL CONTAMINATION



A. FILTER SLUDGE

B. EFFLUENT (METHANOL ELUTION) FROM  
SILICA GEL CHROMA GRAPHY

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FIGURE 8. FREEZE POINTS OF SHALE OIL JP-5 FRACTIONS

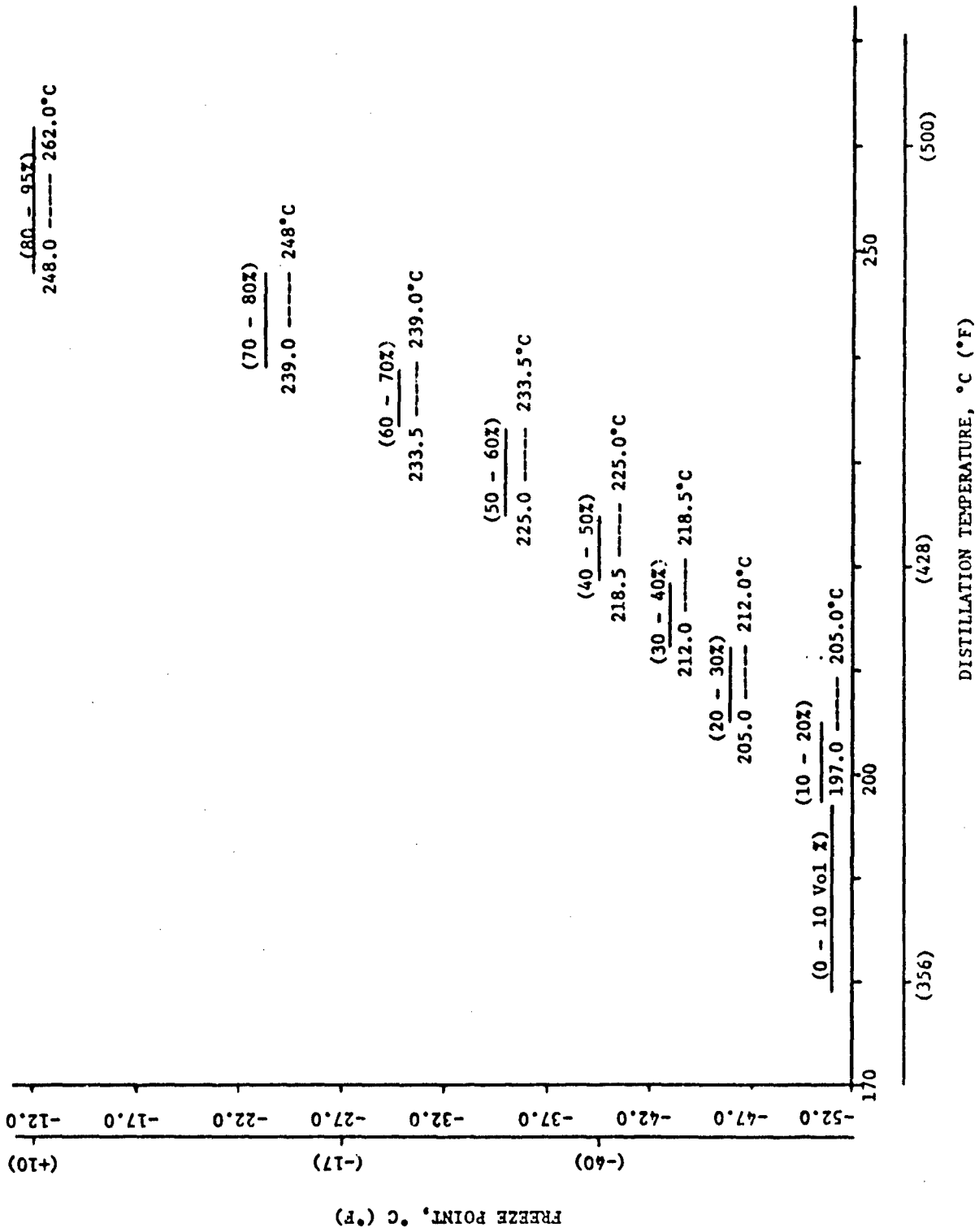


FIGURE 9. VARIATION OF FREEZE POINT WITH DISTILLATION END POINT FOR SHALE OIL JP-5 FRACTIONS

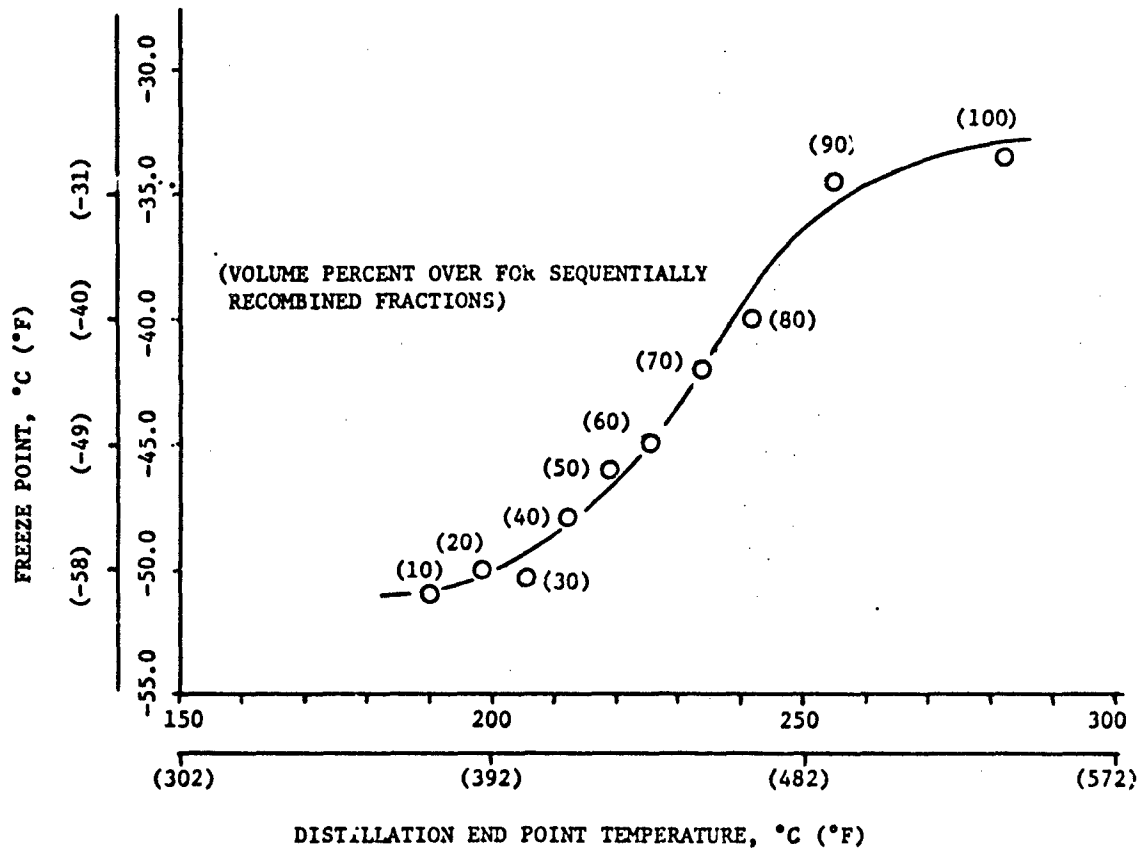


FIGURE 10. EFFECT OF CLAY TREATING ON LIGHT TRANSMITTANCE OF SHALE OIL JP-5

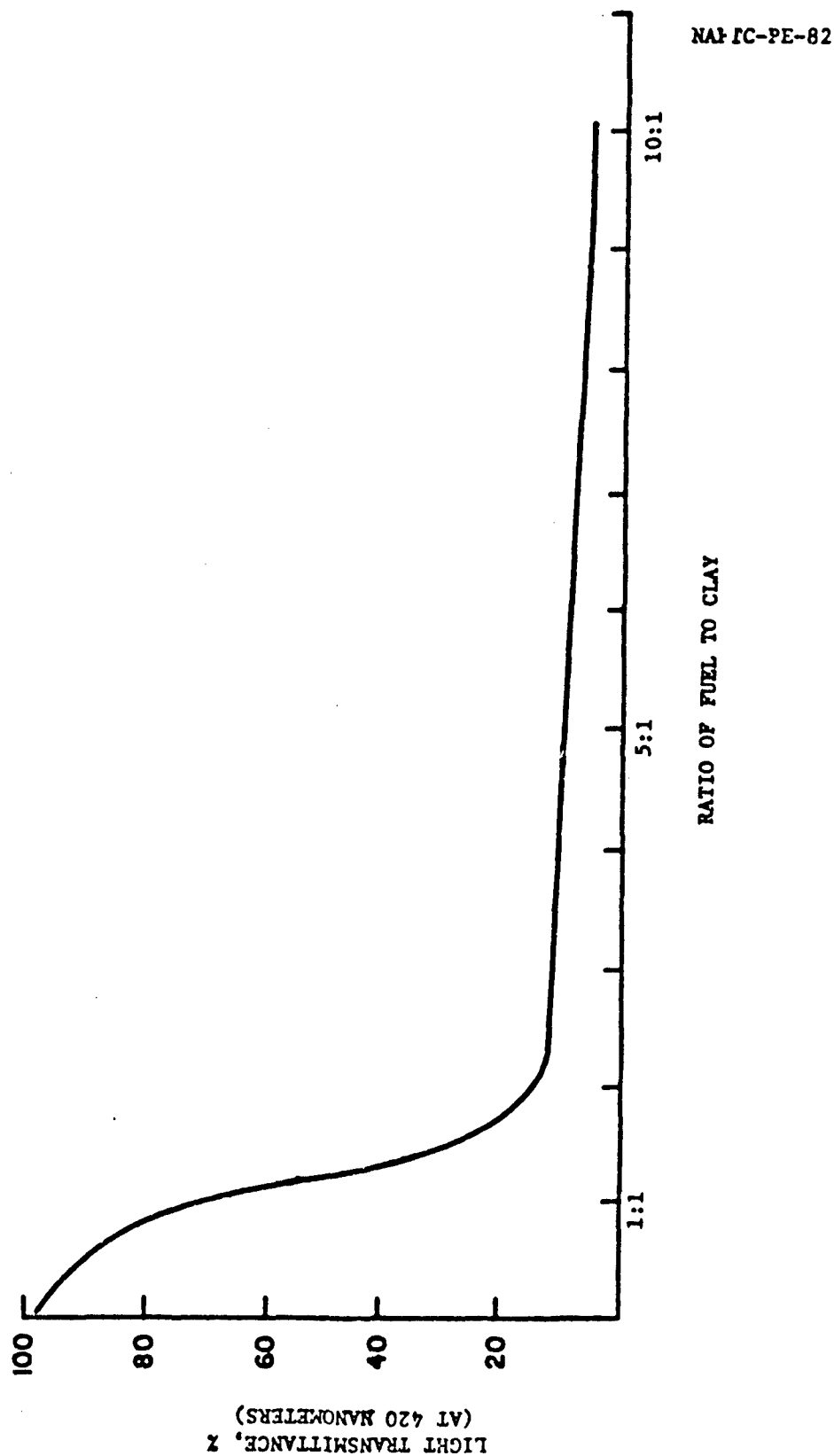




TABLE I  
PERFORMANCE RATINGS AT STANDARD SEA LEVEL STATIC CONDITIONS

Parameter	Take-Off and Military	Normal	Ratings			Ground Start Ground Idle	Flight Autorotation
			90% Normal	75% Normal			
Shaft Power $\text{W} \times 10^5$ (HP)	2.36 (317)	2.01 (270)	1.81 (243)	1.51 (203)		.261 (35) max.	0 (0)
Net Thrust N (lb.), min.	147 (33)	125 (28)	116 (26)	93 (21)		45 (10) max.	45 (10) max.
Gas Producer rpm	860 (51,600)	829 (49,760)	811 (48,650)	783 (46,950)		533 (32,000)	533 (32,000)
Output Shaft rpm	100 (6,000)	100 (6,000)	100 (6,000)	100 (6,000)		75 - 105 (4,500 - 6,300)	98 - 106 (5,900 - 6,360)
Specific Fuel Consumption, $\text{kgW}^{-1}\text{s}^{-1}$ (lb shp <sup>-1</sup> hr <sup>-1</sup> )	$1.178 \times 10^{-7}$ (0.697)	$1.193 \times 10^{-7}$ (0.706)	$1.225 \times 10^{-7}$ (0.725)	$1.286 \times 10^{-7}$ (0.762)		$(7.69 \times 10^{-6} \text{ kg s}^{-1})$ (61 lb/hr)	$(7.69 \times 10^{-6} \text{ kg s}^{-1})$ (61 lb/hr)
Ram Power Rating, Torque at Output Shaft, Nm (lb/ft)	397 (293)	338 (249)	338 (249)	228 (249)		-	-
Measured Rated Gas Temp., °C (°F)	749.0 (1380)	693.5 (1280)	663.5 (1226)	620.0 (1148)		385.0 ± 55.5 (725 ± 100)	385.0 ± 55.5 (725 ± 100)

TABLE II

## LABORATORY ANALYSIS OF PETROLEUM DERIVED JP-5 FOR T63-A-5A ENGINE TEST

	JP-5 Used	Average JP-5 (a)	MIL-T-5624J Requirements	
			Minimum	Maximum
Gravity, Specific 15.5/15.5°C (60/60°F)	0.8114	0.8170	0.788	0.845
Gravity, °API, 15.5/15.5°C (60/60°F)	42.9	41.7	36.0	48.0
Distillation, IBP, °C (°F)	176.5 (350)	-	-	-
5% Over °C (°F)	188.0 (370)	-	-	-
10% Over °C (°F)	192.0 (378)	197.0 (387)	-	204.5 (400)
20% Over °C (°F)	198.0 (388)	-	-	-
30% Over °C (°F)	202.0 (396)	-	-	-
40% Over °C (°F)	208.0 (406)	-	-	-
50% Over °C (°F)	213.5 (416)	216.5 (422)	-	-
60% Over °C (°F)	216.5 (422)	-	-	-
70% Over °C (°F)	223.5 (434)	-	-	-
80% Over °C (°F)	229.0 (444)	-	-	-
90% Over °C (°F)	238.0 (460)	243.0 (469)	-	-
95% Over °C (°F)	245.5 (474)	-	-	-
End Point, °F	258.0 (496)	263.5 (506)	-	288.0 (550)
Recovery % Vol.	98.5	-	-	-
Residue % Vol.	1.0	-	-	1.5
Loss, % Vol.	0.5	-	-	1.5
Gum, Existent, mg/100 ml	0	1.3	-	7
Sulfur, % Wt.	0.06	0.096	-	0.4
F.I.A Saturates, % Vol.	80.86	-	-	-
Olefins, % Vol.	0.95	0.8	-	5.0
Aromatics, % Vol.	18.10	16.0	-	25.0
Aniline Point, °C	61.7	62.5	-	-
Aniline Gravity, Constant	6.139	6.059	4.500	-
Heat of Combustion, MJ Kg <sup>-1</sup> (BTU/lb)	43.170 (18,560)	43.091 (18,526)	42.565 (18,300)	-
Corrosion, Copper Strip	1-a	-	-	1-b
Smoke Point, mm	28	22.2	19	-
Freeze Point, °C (°F)	-50.0 (-58)	-49.0 (-56)	-	-46.0 (-51)
Flash Point, °C (°F)	63.5 (146)	-	60.0 (140)	-
Viscosity, $\text{m}^2 \text{s}^{-1} \times 10^{-6}$ (cgs), 38.0°C (100°F)	1.55	-	-	-
Viscosity, $\text{m}^2 \text{s}^{-1} \times 10^{-6}$ (cgs), -34.5°C (-30°F)	9.40	10.5	-	16.5
Contamination, mg l <sup>-1</sup>	1.80	-	-	1.0
Thermal Stability @ 260.0°C (500°F) (JFTOT)	Pass	Pass	-	Pass
Water Separator Test, Modified	98	94	85	-

(a) Mineral Industry Surveys, Aviation Turbine Fuels, 1973 Reference.

TABLE III  
CORRECTED ENGINE DATA FOR PETROLEUM AND SHALE OIL JP-5 TYPE FUELS

<u>Fuel: JP-5</u>					
<u>Power Setting</u>	<u>T<sub>5</sub> - Corr., °C (°F)</u>	<u>SP, W X 10<sup>5</sup> (HP)</u>	<u>W<sub>f</sub>, Kgs<sup>-1</sup> X 10<sup>-3</sup> (lb/hr<sup>-1</sup>)</u>	<u>SFC, Kgw<sup>-1</sup>S<sup>-1</sup> X 10<sup>-7</sup> (lb shp<sup>-1</sup> hr<sup>-1</sup>)</u>	
Max. Power	672.0 (1242)	1.923 (258)	2.482 (197)	1.291 (0.763)	
Normal Rated Power	623.0 (1153)	1.603 (215)	2.141 (170)	1.336 (0.790)	
90%	599.5 (1111)	1.454 (195)	1.978 (157)	1.360 (0.805)	
60%	517.0 (963)	0.872 (117)	1.424 (113)	1.363 (0.966)	
40%	476.0 (889)	0.454 (61)	1.096 (87)	2.418 (1.426)	
Flight Idle	442.0 (828)	0.254 (34)	0.907 (72)	3.570 (2.118)	
Ground Idle	430.5 (807)	0.164 (22)	0.668 (53)	4.073 (2.409)	
<u>Fuel: Shale Oil JP-5</u>					
<u>Power Setting</u>	<u>T<sub>5</sub> - Corr., °C (°F)</u>	<u>W X 10<sup>5</sup> (SHP)</u>	<u>W<sub>f</sub>, Kgs<sup>-1</sup> X 10<sup>-3</sup> (lb/hr<sup>-1</sup>)</u>	<u>SFC, Kgw<sup>-1</sup>S<sup>-1</sup> X 10<sup>-7</sup> (lb shp<sup>-1</sup> hr<sup>-1</sup>)</u>	
Max. Power	672.0 (1242)	1.925 (258)	2.558 (203)	1.329 (0.787)	
Normal Rated Power	623.0 (1153)	1.588 (213)	2.217 (176)	1.396 (0.826)	
90%	599.5 (1111)	1.423 (191)	2.041 (162)	1.434 (0.848)	
60%	517.0 (963)	0.877 (118)	1.487 (118)	1.695 (1.000)	
40%	476.0 (889)	0.468 (63)	1.159 (92)	2.476 (1.466)	
Flight Idle	442.0 (828)	0.216 (29)	0.932 (74)	4.314 (2.551)	
Ground Idle	430.5 (807)	0.119 (16)	0.668 (53)	5.613 (3.312)	

TABLE IV  
LABORATORY ANALYSIS OF SHALE OIL DERIVED JP-5

	Oil Shale Derived JP-5	Average JP-5 (a)	MIL-T-5624J Requirements	
			Minimum	Maximum
Gravity, Specific 15.5/15.5°C (60/60°F)	0.8058	0.8170	0.788	0.845
Gravity, °API, 15.5/15.5°C (60/60°F)	44.1	41.7	36.0	48.0
Distillation, IBP, °C (°F)	171.1 (340)	-	-	-
5% Over °C (°F)	185.5 (366)	-	-	-
10% Over °C (°F)	191.0 (376)	197.0 (387)	-	204.5 (400)
20% Over °C (°F)	199.0 (390)	-	-	-
30% Over °C (°F)	205.5 (402)	-	-	-
40% Over °C (°F)	212.0 (414)	-	-	-
50% Over °C (°F)	219.0 (426)	216.5 (422)	-	-
60% Over °C (°F)	225.5 (438)	-	-	-
70% Over °C (°F)	233.5 (452)	-	-	-
80% Over °C (°F)	242.0 (468)	-	-	-
90% Over °C (°F)	254.5 (490)	243.0 (469)	-	-
95% Over °C (°F)	265.5 (510)	-	-	-
End Point, °F	282.0 (540)	263.5 (506)	-	288.0 (550)
Recovery % Vol.	97.8	-	-	-
Residue % Vol.	1.0	-	-	1.5
Loss, % Vol.	1.2	-	-	1.5
Gum, Existent, mg/100 ml	81.7	1.3	-	7
Sulfur, % Wt.	0.05	0.096	-	0.4
F.I.A Saturates, % Vol.	71.76	-	-	-
Olefins, % Vol.	2.29	0.8	-	5.0
Aromatics, % Vol.	25.95	16.0	-	25.0
Aniline Point, °C	61.8	62.5	-	-
Aniline Gravity, Constant	6.315	6.059	4.500	-
Heat of Combustion, MJ Kg <sup>-1</sup> (BTU/lb)	43.105 (18,532)	43.091 (18,526)	42.565 (18,300)	-
Corrosion, Copper Strip	1-a	-	-	1-b
Smoke Point, mm	22	22.2	19	-
Freeze Point, °C (°F)	-22.5 (-28)	-49.0 (-56)	-	-46.0 (-51)
Flash Point, °C (°F)	65.5 (150)	-	60.0 (140)	-
Viscosity, m <sup>2</sup> s <sup>-1</sup> X 10 <sup>-6</sup> (cks), -34.5°C (-30°F)	Frozen	10.5	-	16.5
Contamination, mg l <sup>-1</sup>	164.20	-	-	1.0
Thermal Stability @ 260.0°C (500°F) (JFTOT)	Fail	Pass	-	Pass
Water Separator Test, Modified	76	94	85	-

(a) Mineral Industry Surveys, Aviation Turbine Fuels, 1973 Reference.

TABLE V  
REDISTILLATION OF SHALE OIL JP-5 (a)

Test	FUEL PROPERTIES				
	Distillation Temperatures of Runs			Composite of Runs	
	As Received	IBP to 232.0°C (450°F)	(b) 232.0 to 255.5°C (c) (450 to 492°F)	232.0 to 255.5°C (c)	Combined Cuts
Existent Gum, mg/100 ml	81.7	1.4	2.4	1.5	(d)
Basic Nitrogen, ppm	895	1010	666	802	(d)
Thermal Stability, JFTOT, °C (°F)	232.0 (450), Pass	-	-	260.0 (500), Pass	

30

(a) Shale Oil JP-5 was redistilled at atmospheric pressure through all-glass apparatus.

(b) Distillate Yield = 60 percent (volume) of initial fuel.

(c) Distillate Yield = 30 percent (volume) of initial fuel.

(d) Calculated value.

	As Received	Ratio of Fuel to Clay			(b) Last 10% Out 9:1	Total Composite	Requirement
		First 10% 1:1	2:1	2.2:1			
Contamination, $\text{mg l}^{-1}$	164.2	0	-	-	5.8	-	1
Existent Gum, mg per 100 ml	81.7	40.6	-	-	86	39.6	7
Thermal Oxidation Stability, °C (°F) (JFTOT)	232.0 (450) Pass	251.5 (485) Pass	-	-	240.5 (465) Pass	246.0 (475) Pass	260.0 (500) Pass
Basic Nitrogen Content, ppm	895	0	44	453	890	780	-
Soluble Metals, ppb							
Cu	240	0	-	-	0 - 5	-	-
Fe	0	0	-	-	0	-	-
Zn	55	0	-	-	0	-	-

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(a) 10 liters of shale oil JP-5 percolated through one liter of clay (see Description Section).

(b) There was a hold-up of one liter in the apparatus.

TABLE VII  
ANALYSIS OF CLAY TREATED SHALE OIL JP-5

CONDITIONS: (170 l (45 gallons) of shale oil JP-5 recycled five times through 16.1 l (0.57 ft<sup>3</sup>) of clay (10.9 Kg (24 lb) clay).

First Treatment: 9.2:1 volume ratio of fuel to clay.

	<u>Percent Light Transmission (a)</u>
Before Treatment	0
1st Pass	3
2nd Pass	4.5
3rd Pass	0.0 (probably due to back flushing)
4th Pass	4.0
5th Pass	4.2
Existent Gum, mg/100 ml	54.9 (total composite) <sup>(b)</sup>
Thermal Stability, JFTOT, °C (°F)	240.5 (465) Pass <sup>(b)</sup>

Second Treatment: 4.6:1 volume ratio of fuel to clay.

1st Pass	10.8
2nd Pass	6.5
3rd Pass	9.0
4th Pass	8.0
5th Pass	8.2
Existent Gum, mg/100 ml	64.4 <sup>(b)</sup>
Basic Nitrogen, ppm	530 <sup>(b)</sup>
Thermal Stability, JFTOT, °C (°F)	251.5 (485) Pass <sup>(b)</sup>

NOTE: (a) measured on a Bausch and Lomb Spectronic 20 spectrophotometer at 540 nm.  
(b) fuel which was obtained after 5th pass through clay was analyzed.

TABLE VIIIEFFECTS OF ACID TREATMENT ON SHALE OIL JP-5

TREATMENT LEVEL: 0.90 Kg (2 lb) of 98 percent  $H_2SO_4$  to 1 barrel of fuel.

<u>Fuel Properties</u>	<u>Before Acid Treatment (a)</u>	<u>After Acid Treatment</u>		<u>MIL-T-5624J Requirement</u>
		<u>No Clay</u>	<u>Clay</u>	
Basic Nitrogen, ppm	890	0	0 (530)(b)	None
Existent gum, mg/100 ml	81.7	46	39.4 (64.4)(b)	7
Thermal Stability JFTOT at 251.5°C, (485°F)	Fail	Pass	Pass (Fail)(b)	Pass at 260.0°C (500°F)
Aromatics % by Vol.	26	24.5	24.8	25 max.
Olefins % by Vol.	2.3	1.04	1.29	5 max.
Freeze Point, °C (°F)	-33.5 (-28)	-31.0 (-24)	-31.0 (-24)	-46.0 (-51)

NOTE: (a) Data refer to the as received shale oil JP-5.

(b) Data in brackets were obtained on clay treated shale oil JP-5 before acid treatment.



TABLE IX  
N-PARAFFINS IN SHALE OIL JP-5 (a)

<u>Component</u>	<u>Weight Percent</u>
<u>n</u> -nonane	0.90
<u>n</u> -decane	3.28
<u>n</u> -undecane	7.46
<u>n</u> -dodecane	7.12
<u>n</u> -tridecane	6.66
<u>n</u> -tetradecane	5.14
<u>n</u> -pentadecane	3.32
<u>n</u> -hexadecane	2.45
TOTAL	36.33

NOTE: (a) Determined by Dr. R. N. Hazlett, Naval Research Laboratory, by glpc on a capillary column using electronic integration (limits for identification of n-paraffin retention time: +0.5 min).

TABLE XPHYSICAL PROPERTIES OF UREA EXTRACTED SHALE OIL JP-5Test

## ASTM Distillation, D-86

IBP, °C (°F)	60.0 (140) (a)
10%	193.5 (380)
50%	220.0 (428)
95%	266.5 (512)
End Point	266.5 (512)
Recovery, %	96.1
Residue, %	2.4
Loss, %	1.5

## Hydrocarbon Type, FIA

Saturates, Vol %	72.02
Aromatics, Vol %	27.98

Freeze Point, °C (°F)	-48.0 to -52.0 (-54 to -62) (b), (c)
Viscosity, $\text{m}^2 \text{s}^{-1} \times 10^{-6}$ , (cks), (-18°C (0°F))	5.07 (5.07) (b)
Viscosity, $\text{m}^2 \text{s}^{-1} \times 10^{-6}$ , (cks), (-34.5°C (-30°F))	9.48 (9.48) (b)
Flash Point, °C (°F) (Seta Flash)	63.5 (146)

NOTE: (a) The low initial boiling point is caused by traces of petroleum ether remaining in the fuel after urea extraction (see Description).

(b) The first three ml of distillate from this run were discarded and the Flash Point, Freeze Point and Viscosity determined on the remaining distillate.

(c) Differences from separate experiments are thought to be within experimental error for determination of freeze points.

TABLE XI

ANALYSIS OF UREA EXTRACT FROM SHALE OIL JP-5

<u>n-Paraffins</u>	<u>Normalized Weight Percent Yield (a)</u>	<u>Absolute Yield, g (b)</u>	<u>Percent Extracted From Original Shale Oil JP-5 (c)</u>
<u>n-Pentane (C<sub>5</sub>)</u>	1.28	-	-
<u>n-Nonane (C<sub>9</sub>)</u>	0.96	0.17	18.9
<u>n-Decane (C<sub>10</sub>)</u>	3.86	0.69	21.0
<u>n-Undecane (C<sub>11</sub>)</u>	11.54	2.07	27.7
<u>n-Dodecane (C<sub>12</sub>)</u>	16.50	2.96	41.5
<u>n-Tridecane (C<sub>13</sub>)</u>	19.13	3.43	50.5
<u>n-Tetradecane (C<sub>14</sub>)</u>	18.71	3.36	65.3
<u>n-Pentadecane (C<sub>15</sub>)</u>	15.16	2.72	81.9
<u>n-Hexadecane (C<sub>16</sub>)</u>	9.47	1.70	69.3
<u>n-Heptadecane (C<sub>17</sub>)</u>	<u>1.38</u>	0.24	(e)
TOTAL	97.99 (d)		

NOTE: (a) Determined by R. N. Hazlett, Naval Research Laboratory, by glpc on a capillary column using electronic integration.

(b) Obtained by multiplying the normalized weight percent yield of extracted paraffin by the total amount extracted (17.97 g).

(c) Based on values reported in Table IX.

(d) Remaining 2 percent of material includes i-alkenes and 2-methyl alkanes of similar carbon number to the n-paraffins. Thus, urea extraction procedure is highly selective in removing n-paraffins.

(e) Not determined for original Shale Oil JP-5.

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